

MOISTURE CONTENT AND FLAVOR STABILITY OF BATCH VACUUM FOAM-DRIED WHOLE MILK

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ABSTRACT

Fifteen lots of vacuum foam-dried whole milk, ranging in moisture from 1.1-5.1%, were nitrogen-packed in cans containing 0.3-1.1% oxygen in the headspace gas and stored at 73 F. Samples were taken from storage at intervals and analyzed for flavor, moisture, TBA number, and headspace oxygen over a period of 26 wk. The time in storage before the first significant change in flavor could be detected was found to increase five- to sixfold when the moisture content of the dry milk was above about 2.8%. A statistical monomolecular layer of water (BET monolayer) occurred at a moisture content of 3.95%. In-package desiccation resulted in reduced flavor stability.

Several investigators have shown the influence of moisture content on the flavor stability of spray dried whole milk when some oxygen is present in the package. For example, Findlay et al. (3) found that flavor stability was optimized at a moisture content of 3.3%, and Holm et al. (4) reported that the best stability against oxidation was obtained with a moisture content between 2 and 3%. In addition, the latter workers pointed out that since no two powders manufactured by different processes are alike with respect to their water adsorption capacities, each powder would have its own optimum. Tamsma and Pallansch (9) reported on some factors affecting the storage properties of vacuum foam-dried whole milk prepared by the method of Sinnamon et al. (8). The adsorption theory of Brunauer et al. (1) has been used successfully to predict optimum moisture content of many foodstuffs (6, 7). This paper reports the effect of moisture content on the flavor stability of batch vacuum foam-dried whole milk stored at 73 F and its moisture content at a statistical monomolecular layer (BET monolayer) of adsorbed water.

EXPERIMENTAL PROCEDURES

Preparation of the samples. The whole milk used in these studies was Grade A market milk procured during spring and summer months from a large dairy in Philadelphia. It had been pasteurized (162 F, 16½ sec) but not forewarmed. Drying was according to the procedure of Sinnamon et al. (8) in which a foamed concentrate is batch-dried under vacuum. Nine batches were dried to different moisture levels in the range of 1.10 to 5.11%.

The products were nitrogen-packed in hermetically sealed cans averaging 0.5% by volume residual oxygen content corresponding to 0.023 ml of O₂ per gram of dry milk. This was accomplished by filling the cans with product and sealing with a lid containing a hole 0.04 in. diameter. They were then evacuated in a chamber maintained at 1 mm Hg absolute pressure for one-half hour. The vacuum was released with virtually pure nitrogen gas and the holes sealed with solder.

To hold constant the influence of variables other than moisture content, six of the nine batches were divided and packaged with and without an in-package desiccant (IPD). The desiccant was anhydrous calcium oxide pellets packed in heat-sealed, sift-proof paper envelopes. The ratio of dry milk to desiccant was 6.5 to 1. Storage was conducted at 73 F.

Tasting procedure. The samples were tasted at 2-wk intervals for 26 wk. Sensory analysis was by the Rank Paired Comparison Test, as described by Terry et al. (10). In this test, a freshly dried control sample and two stored samples were compared by presenting them in all combinations as pairs to a taste panel of 10 to 20 trained judges. The pairs were presented separately. The judges were asked, "Which sample of each pair tastes more like fresh milk?" The one tasting more like fresh was scored 1 and the other 2. Since each sample was tasted twice, the sum of the scores from each judge would be either 2, 3, or 4. Valid comparisons between any two samples were obtained by the analysis of variance. The results are reported as per cent significant level of difference (SLD) between the fresh control and each stored sample, or between the two stored samples. SLD = 5% or less ($P \leq 0.05$)

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is considered a real difference. Products prepared for use as fresh controls were equal in initial flavor regardless of moisture content.

Chemical analyses. Each time a stored sample was tasted, it was analyzed for moisture by the Karl Fischer method, and for headspace oxygen by a Beckman Model C² analyzer. TBA analyses were made on about half of the powders tasted. The method of Dunkley and Jennings (2) was employed.

BET monolayer determination. Small samples of dry whole milk were exposed in desiccators to constant humidities to determine the BET isotherm. The desiccator bottom in each case contained a saturated salt solution, with an excess of undissolved salt which yielded constant humidities of 12.0, 22.5, 33.4, and 43.7% at 73 F (5, 12). A well-blended batch of dry whole milk was weighed into weighing jars, 0.2 to 0.5 g per jar. An additional sample was analyzed for moisture content by the Karl Fischer method. Two jars were placed in each desiccator (i.e., at each humidity) and held at 73 F until constant weight was reached. In preliminary tests, seven to ten days were required to reach constant weight. For the equilibrium moisture determinations, all jars were weighed after ten to fourteen days of exposure. Weight change permitted calculation of the equilibrium moisture content.

Four batches of dry whole milk were used to determine the sorption isotherms. The modified method of Salwin (7) was then applied to determine the moisture content corresponding to the BET monolayer.

RESULTS AND DISCUSSION

Table 1 gives the time when the significant level of difference between the fresh control and

¹ Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

TABLE 1
Effect of moisture content on flavor stability*

| Batch number | Moisture content (%) | Weeks to reach SLD = 5.0% or less |
|--------------|----------------------|-----------------------------------|
| 1 | 1.10 | 2 |
| 2 | 1.13 | 4 |
| 3 | 1.27 | 2 |
| 4 | 2.41 | 2 |
| 5 | 2.59 | 4 |
| 6 | 2.84 | 13 |
| 7 | 3.23 | 10 |
| 8 | 4.39 | 13 |
| 9 | 5.11 | 10 |

* In-package desiccation was not used.

the stored sample in each batch fell below 5.0%. It demonstrates that there exists a moisture content between 2.59 and 2.84% below which flavor stability is materially decreased under conditions that prevailed during these tests.

If only the results given in Table 1 were obtained in this work, it could be said that the minimum moisture level for optimum stability was possibly a consequence of differences among milks of the various batches, and among the time-temperature drying conditions used to attain the various initial moisture contents. However, when the results of the batches with and without IPD are compared, it can be seen that this possibility was not dominant, and that moisture level was definitely the factor that influenced flavor stability. For these comparisons, there were no differences in the respective dry milks at the start of storage. Table 2 was compiled by combining the results of all of the taste tests which each batch had undergone and applying the usual statistical analysis to the rankings. For example, Batch 2 was judged 155 times during the life of the test. The freshly dried control averaged 2.15 across the test, the material without IPD averaged 3.35 and with IPD, 3.50. In all cases the half of the batch that contained IPD had poorer storage stability. Moreover, for each batch except 1 and 3, this difference was significant ($P < 0.05$). Furthermore, it will be noted that, in general, less difference was observed at the lower initial moisture contents.

In 39 of 42 stored pairs tested, the TBA number was higher for the IPD half-batch. In two of the remaining three, the values were the same. These numbers ranged from .040 to .080. While TBA values are not advanced as conclusive, this provides strong support for the preceding observation that oxidative deterioration was more evident in the sample with lower moisture.

Figure 1 illustrates the rate at which the in-package desiccant reduced the moisture content. Even the highest initial moisture content was reduced below 2.59% within about 1 wk, and since the first taste test was held after 2 wk of storage, results obtained with IPD are further evidence that there is a minimum moisture level for better flavor life under the storage conditions of these tests.

Figure 2 represents the averaged equilibrium moisture as a function of relative humidity. The moisture contents equivalent to the BET monolayer were found to be 3.821, 4.405, 3.897, and 4.393 g per 100 g of solids, averaging 4.12 g per 100 g, or 3.95% moisture. This value is well within the range found by taste tests to

TABLE 2

Cumulative taste panel results showing effect of IPD on flavor stability

| Batch number | Initial moisture (%) | Number taste tests | Number judgments | Avg ranking scores* | | | SLD** (%) |
|--------------|----------------------|--------------------|------------------|---------------------|-------------|----------|-------------------|
| | | | | Freshly dried | Without IPD | With IPD | |
| 1 | 1.10 | 8 | 86 | 2.14 | 3.40 | 3.47 | Between 50 & 40 |
| 2 | 1.13 | 11 | 155 | 2.15 | 3.35 | 3.50 | Between 5 & 2.5 |
| 3 | 1.27 | 8 | 78 | 2.13 | 3.42 | 3.45 | Between 100 & 50 |
| 4 | 2.41 | 11 | 186 | 2.17 | 3.25 | 3.59 | Less than 0.1 |
| 6 | 2.84 | 11 | 121 | 2.31 | 3.14 | 3.55 | Less than 0.1 |
| 7 | 3.23 | 12 | 176 | 2.26 | 3.25 | 3.49 | Between 0.5 & 0.1 |

** Comparing the half-batch stored without IPD and that stored with IPD. In every case, the half-batch stored without IPD was judged to be more stable.

* Avg ranking scores = $\frac{\sum \text{individual judges scores}}{\text{number of judgments}}$

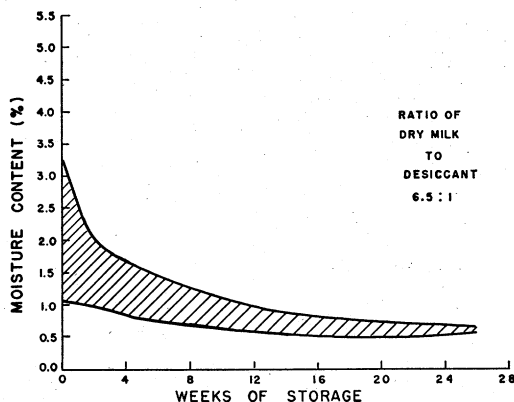


FIG. 1. Moisture reduction by in-package desiccant at 73 F.

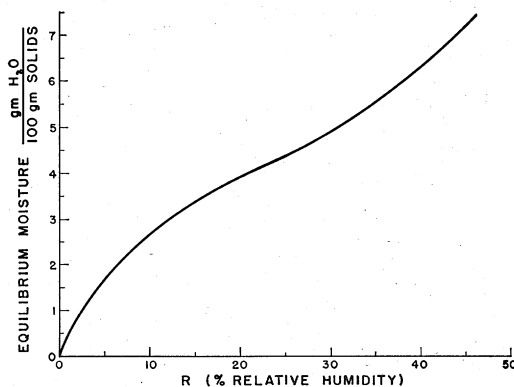


FIG. 2. Sorption isotherms of vacuum foam-dried whole milk at 73 F.

be optimum for flavor stability. Thus, the greater flavor stability of the samples above 2.8% moisture may be explained on the basis of the theory proposed by Salwin (6), that the monomolecular layer of moisture acts as a protective covering for the dry milk. The work of Uri (11) may also help to explain the results

found here. He connected the concentration of water as it influences metal ion catalysis with the abrupt decrease in the rate of oxidative reactions above a critical moisture content in dehydrated foods.

The range of oxygen contents for over 500 cans used in this study was 0.3 to 1.1% by volume, averaging 0.5%. This range was a random variation among the cans within each batch and across all batches. It is attributable to nonuniformity inherent in gassing technique and to experimental error in the analysis. No effect on flavor stability was apparent because of variation in headspace oxygen. Such influence that it might have had was completely overshadowed by the influence of moisture content.

CONCLUSIONS

Although the problem of attaining adequate flavor shelf life at room temperature may not be solved by control of moisture content, this study shows that storage stability is markedly influenced by moisture when oxygen is present even in small amounts. The optimum range of moisture content for vacuum foam-dried whole milk stored at 73 F in cans under N_2 with 0.3 to 1.1% O_2 appears to lie above about 2.8%.

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